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PATENT ABSTRACTS OF JAPAN

(11)Publication number : 63-150215

(43)Date of publication of application : 22.06.1988

(51)Int.Cl. A61K 7/11

(21)Application number : 61-296176

(71)Applicant : KAO CORP

(22)Date of filing : 12.12.1986

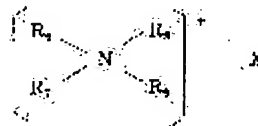
(72)Inventor : AKIMOTO KEIICHI
ANDO YOJI

(54) HAIR COSMETIC

(57)Abstract:

PURPOSE: To obtain a hair cosmetic extremely stable with time and exhibiting excellent hair-setting effect without degrading the feeling of hair by applying to hair and rinsing with water, by using an ampholytic polymer, a water-soluble inorganic salt and xanthan gum.

CONSTITUTION: The objective cosmetic contains (A) 0.1W20wt.% ampholytic polymer insoluble in water and soluble in 10% salt solution as 0.1wt.% at 20°C, e.g. a copolymer of an acidic vinyl monomer (e.g. acrylic acid) and a basic vinyl monomer (e.g. diethylaminoethyl methacrylate), (B) 0.1W30wt.% water-soluble inorganic salt (e.g. sodium sulfate, potassium chloride, etc.) and (C) 0.1W5wt.% xanthan gum. A good conditioning effect can be attained by further compounding 0.1W10wt.% cationic surfactant of formula (one or two of R₆WR₉ are 8W22C alkyl or hydroxyalkyl and the remainders are 1W3C alkyl, benzyl, etc.; X is halogen or 1W2C alkylsulfuric acid residue).



LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

Japan Patent Office
Public Patent Disclosure Bulletin

Public Patent Disclosure Bulletin No.: 1-190620
Public Patent Disclosure Bulletin Date: July 31, 1989
Request for Examination: Not yet made
Number of Inventions: 1
Total Pages: 12

| Int. Cl. ⁴ | Identification Code | Internal File Nos. |
|-----------------------|---------------------|--------------------|
| A 61 K 7/06 | | 7430-4C |
| 7/08 | | 7430-4C |

Title of Invention: Hair cosmetic
Patent Application No.: 63-14295
Patent Application Date: January 25, 1988
Inventor: Keiichi Akimoto
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shi, Chiba Pref.
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Tokyo
Agent: Mitsuyuki Ariga, Patent Attorney (and 2
others)

Specifications

1. Title of Invention:

Hair cosmetic

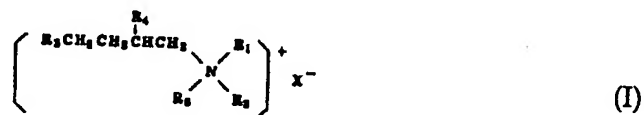
2. Claims:

1. A hair cosmetic which contains the following ingredients (A), (B), and (C):

(A) an amphoteric polymer soluble in a 10 wt % aqueous solution of sodium chloride and insoluble in water at a concentration of 0.1 wt % at 20°C
0.1-20 wt %

(B) a water-soluble inorganic salt
0.1-30 wt %

(C) 1 or 2 or more branched quaternary ammonium salts shown by formula (I) below
0.01-20 wt %



(wherein R_1 and R_2 are benzyl groups or C_{1-3} alkyl or hydroxyalkyl groups; R_3

and R_4 are C_{2-16} alkyl groups; R_5 is a group shown by $R_2CH_2CH_2CHCH_2-$ or a C_{1-3} alkyl group; and X is a halogen ion or organic anion).

3. Detailed Explanation of Invention:

Industrial Field of Application

This invention concerns a hair cosmetic. In particular, it concerns a hair cosmetic which contains an amphoteric polymer, a water-soluble inorganic salt, and a branched quaternary ammonium salt, which shows an excellent hair setting performance by rinsing with water after it is applied to the hair, and which has little oily feel and excellent smoothness, softness, and anti-static effectiveness, without detriment to the feeling of the hair.

Prior Art and Problems in It

The styling of hair is one of the most important aspects of cosmetology, and various kinds of cosmetic processes are used for this purpose. For example, one temporary method is to wind the hair in curlers and dry it, and to style the hair using a dryer and brush. Hair cosmetics, including setting lotions, blowing agents, hair sprays, etc., are used in order to make the hair styling easier to perform and to hold the finished hair style. Furthermore, polymers which are soluble in water or organic solvents such as alcohols are added to the hair cosmetics used for this purpose, for example, polyvinyl pyrrolidone, vinyl pyrrolidone/vinyl acetate copolymers, vinyl pyrrolidone/vinyl acetate/alkylamino acrylates, methyl vinyl ether/maleic anhydride copolymers, vinyl acetate/crotonic acid copolymers, copolymers of acrylic or methacrylic acid and acrylic or methacrylic esters. Hair styles have been formed and held in shape by applying these substances to the hair or adhering them as aerosols.

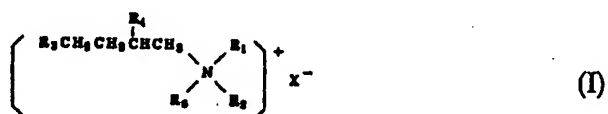
However, conventional hair cosmetics have had the drawbacks that they require that large quantities of polymers be adhered to the hair in order to hold the hair styles, and because the surface tensions of these polymers adhered to the hair are higher than the critical surface tension of the hair, they form small lumps and adhere to the hair as islands, so that the feel of the hair is made very rough and it is difficult to comb.

Means of Solving the Problems

In this situation, the inventors performed careful research, as a result of which they discovered that if a hair cosmetic containing specific amphoteric polymers and water-soluble inorganic salts is used, excellent style holding and forming ability can be obtained by rinsing after the cosmetic is applied to the hair. An application for a patent was previously made (Japan Patent Disclosure Bulletin No. 62-174004). In the present application, the inventors discovered that, if specific branched quaternary ammonium salts are combined with the aforementioned ingredients, the feel of the hair can be improved without detriment to the hair setting performance. Furthermore, especially noteworthy points are that the moistened hair can be quickly dried, it can be set quickly into the desired hair styles, and damage to the hair due to the heat of the dryer during the setting process can be reduced compared with previously developed cosmetics. Thus, they perfected this invention.

That is, this invention provides a hair cosmetic which contains the following ingredients (A), (B), and (C):

- (A) an amphoteric polymer soluble in a 10 wt % aqueous solution of sodium chloride and insoluble in water at a concentration of 0.1 wt % at 20°C
0.1–20 wt %
- (B) a water-soluble inorganic salt
0.1–30 wt %
- (C) 1 or 2 or more branched quaternary ammonium salts shown by formula (I)
below
0.01–20 wt %



(wherein R_2 and R_3 are benzyl groups or C_{1-3} alkyl or hydroxyalkyl groups; R_4 and R_5 are C_{2-16} alkyl groups; R_5 is a group shown by or a C_{1-3} alkyl group; and X^- is a halogen ion or organic anion).

The amphoteric polymers of ingredient (A) used in this invention can be produced by copolymerizing acidic vinyl monomers and basic vinyl monomers, by polymerizing amphoteric monomers, or by introducing acidic groups, basic groups, both acidic and basic groups, or amphoteric groups into synthetic or natural polymers, according to the properties of these polymers. In any case, it is desirable for the positive charge to be almost 0 and for the molecular weight to be in the range of 500–5,000,000, preferably 5,000–500,000.

The following are typical examples of the amphoteric polymers used in this invention.

(1) Copolymers of acidic and basic vinyl monomers

Typical examples of these are amphoteric copolymers which are obtained by copolymerizing monomer mixtures which consist of 45–55 mol % amphoteric vinyl monomers or the salts and 45–55 mol % basic vinyl monomers or their salts in the presence of publicly known accelerants or without these accelerates, at 150°C. The molar percentages mentioned above refer to the case in which the respective vinyl monomers have 1 acidic or basic group in their molecules; if these monomers have multiple acidic or basic groups per molecule, the molar percentages are adjusted in such a way that the positive charges are almost 0.

The acidic vinyl monomers mentioned above are compounds which have acidic groups, such as carboxyl, sulfonic acid, phosphoric acid, etc., groups and polymerizable vinyl groups in their molecules. For example, one can use unsatur-

ated monobasic acids, such as acrylic acid, methacrylic acid, crotonic acid, vinylbenzoic acid, 2-acrylamide-2-methylpropane sulfonic acid, styrene sulfonic acid, vinyl sulfonic acid, allyl sulfonic acid, methallyl sulfonic acid, or 3-methacrylpropane sulfonic acid, or unsaturated dibasic acids, such as itaconic acid, maleic acid, or fumaric acid, or monoesters, etc., of these acids. Furthermore, their salts may be sodium, potassium, or ammonium salts, etc.

The basic vinyl monomers mentioned above are compounds which have basic groups, such as primary, secondary, or tertiary amino groups, and polymerizable vinyl groups in their molecules. For example, one can use dimethylaminoethyl methacrylate, diethylaminoethyl methacrylate, dimethylaminoethyl acrylate, diethylaminoethyl acrylate, dimethylaminopropyl acrylate, dimethylaminopropyl methacrylamide, dimethylaminopropyl acrylamide, 2-vinylpyridine, 4-vinylpyridine, dimethylallylamine, diallylmethylamine, etc., as well as their quaternized derivatives, such as hydrogenated, methylated, or ethylated derivatives. The counter ions may be chlorine, bromine, or other halogen ions, hydroxyl ions, or methylsulfuric acid, etc., groups.

The copolymerization reaction may be performed by methods which are already publicly known, e.g., block polymerization, aqueous solution polymerization, reversed phase suspension polymerization, precipitation polymerization, etc. The reaction temperature may be in the publicly known range in which radical polymerization is ordinarily performed, but in general it is performed without difficulty in the range of 20–150°C in the presence of a radical polymerization initiator.

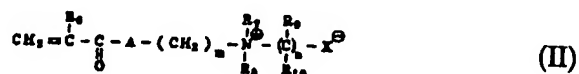
Publicly known radical polymerization initiators may be used, e.g., sodium persulfate, potassium persulfate, ammonium persulfate, 2,2'-azobis(2-amidinopropane) dihydrochloride, benzoyl persulfate, hydrogen persulfate, sodium peracetate, cumene hydroperoxide, azobisisobutyronitrile, etc. Moreover, one can use, for example, sodium hydrogen sulfite, sodium thiosulfate, or ammonium ferrous sulfate as polymerization initiators and accelerants. The quantity of the radical polymerization initiator used varies with the kind used, but in general it should be about 0.01–5 wt % of the total monomer content.

In this polymerization, besides the acidic and basic vinyl monomers, other vinyl monomers which are copolymerizable with acidic and basic vinyl monomers may be included as optional 3rd ingredients, but their content must be 60 mol % or less with respect to the total monomers. These vinyl monomers are monovinyl compounds which are polymerizable by radical polymerization initiators; for example, one can use acrylic esters, such as methyl acrylate, ethyl

acrylate, etc.; methacrylic esters, such as methyl methacrylate, ethyl methacrylate, etc.; styrene compounds, such as styrene, α -methylstyrene, etc.; and acrylamide, methacrylamide, vinyl ether, vinyl acetate, etc.

(2) Polymers of amphoteric monomers

Typical examples of these are those obtained by polymerizing the amphoteric monomers shown by general formula (II) below in a temperature range of 20–120°C, in the presence of a radical polymerization initiator.



(wherein R_6 , R_7 , and R_{10} are hydrogen atoms or methyl groups; R_8 and R_9 are methyl or ethyl groups; A is -O- or -NH-, X is -CO₂-, -SO₂-, or -PHO₂-; and m and n are integers in the range of 1–3).

The amphoteric monomers shown by general formula (II) can be synthesized by reactions of suitable aminoalkyl esters or aminoalkylamides of acrylic or methacrylic acid with lactone, saltone,¹ or cyclic phosphites.

Examples of these compounds are, for example, 3-dimethyl(methacroylox-ethyl)ammonium propane sulfonate, 3-dimethyl(methacrylamide propyl)ammonium propane sulfonate, etc.

The polymerization method used may be a method which is already publicly known, for example, block polymerization, aqueous solution polymerization, precipitation polymerization, etc. The polymerization can be performed without difficulty in the temperature range of 20–150°C in the presence of a radical polymerization initiator.

Examples of these radical polymerization initiators are sodium persulfate, potassium persulfate, ammonium persulfate, 2,2'-azobis(2-amidinopropane) dihydrochloride, benzoyl persulfate, hydrogen persulfate, sodium peracetate, cumene hydroperoxide, azobisisobutyronitrile, etc. The quantity of the radical polymerization initiator used varies with the kind used, but in general it should be about 0.01–5 wt % of the total monomer content.

The quantity of the amphoteric polymer of ingredient (A) in the composition of this invention is 0.1–20 wt %, preferably 0.5–5 wt %, of the total composition.

¹Original: "saruton" - English spelling could not be confirmed. - Translator's note

tion. If it is less than 0.1 wt %, sufficient effect will not be obtained, and if it exceeds 20 wt %, it will be difficult to obtain a homogeneous composition, which is not desirable.

Examples of the water-soluble inorganic salts of ingredient (B) which are used in this invention are alkali metal salts, alkaline earth metal salts, or aluminum salts of hydrochloric acid, sulfuric acid, nitric acid, etc. Among these inorganic salts, preferable ones are potassium sulfate, sodium sulfate, magnesium sulfate, aluminum sulfate, potassium nitrate, sodium nitrate, magnesium nitrate, calcium nitrate, aluminum nitrate, potassium chloride, sodium chloride, magnesium chloride, calcium chloride, aluminum chloride, potassium carbonate, sodium carbonate, and aluminum carbonate, especially sodium sulfate, potassium nitrate, sodium nitrate, potassium chloride, and sodium chloride.

The quantity of the water-soluble inorganic salt of ingredient (B) in the composition of this invention is 0.1–30 wt %, preferably 1–10 wt %, of the total composition. If it is less than 0.1 wt %, the amphoteric polymer will not dissolve easily, and if it exceeds 30 wt %, the salt will remain when [the hair] is rinsed, harming the feel of the hair, and therefore this is not desirable.

The branched quaternary ammonium salt of ingredient (C) of this invention is shown by formula (I); ordinarily, it can be synthesized by using a C_{8-36} Guerbet

alcohol ($R_1CH_2CH_2\overset{R_2}{\underset{|}{C}}HCH_2OH$) as the raw material. Desirable examples of these branched quaternary ammonium salts are monoalkyl-type quaternary salts, such as alkyltrimethylammonium salts, alkyl dimethylhydroxyethylammonium salts, alkyl dimethylbenzylammonium salts, etc., with alkyl groups derived from Guerbet alcohols, and dialkyl-type quaternary salts, such as dialkyl dimethylammonium salts, dialkyl methylhydroxyethyl ammonium salts, dialkyl methylbenzylammonium salts, etc. The counter ions of these ammonium salts may be halogen ions, such as iodine, bromine, etc., or organic anions, such as methosulfate, ethosulfate, methophosphate, ethophosphate, etc., anions. Examples of the alkyl groups derived from Guerbet alcohols are 2-hexyldecyl, 2-octyl dodecyl, 2-decyl tetradecyl, 2-dodecyl hexadecyl, etc. Especially desirable specific examples of these branched quaternary ammonium salts are 2-decyl tetradecyl trimethyl ammonium chloride, 2-dodecyl hexadecyl trimethyl ammonium chloride, di-2-hexyldecyl dimethyl ammonium chloride, d-2-octyl dodecyl dimethyl ammonium chloride, etc. When these compounds are used as hair rinses, the hair is especially smooth to the touch when it is dried.

The branched quaternary ammonium salt of ingredient (C) may be one of the ones mentioned above, or a combination of 2 or more of them.

The quantity of ingredient (C) in the composition is 0.01–20 wt %, preferably 0.1–5 wt %, of the total composition. If it is less than 0.01%, it is difficult to obtain good smoothness, and if it exceeds 20 wt %, the sticky feeling is increased, and the feel of the hair is harmed.

Furthermore, quaternary ammonium salts other than those mentioned above, e.g., branched quaternary ammonium salts synthesized by using cetyltrimethyl ammonium chloride, stearyltrimethyl ammonium chloride, or C_{8-16} oxo alcohols as raw materials, can be used as auxiliary ingredients.

The medium of the hair cosmetic of this invention may be essentially water or an aqueous alcohol solution containing a small quantity of a lower alcohol. If necessary, the pH may be adjusted to 3–11, preferably 5–9, using an acid, alkali, or buffering agent. At this time, it is essential to adjust the pH so that the amphoteric polymer is completely dissolved in the original solution of the hair cosmetic, and when it is diluted, [the polymer] becomes insoluble and precipitates out. That is, if an amphoteric polymer with a positive charge of almost 0 dissolves in a markedly acidic or alkaline medium when the pH is 7, it becomes an essentially cationic or anionic polymer; therefore, the polymer does not precipitate even when it is diluted, and a setting effect is not obtained.

The preparation of the hair cosmetic is not particularly limited; it may be a transparent liquid, lotion, emulsion, foam aerosol (mousse), etc. When it is made a mousse, fluorocarbons, liquefied petroleum gases, dimethylether, etc., may be used, individually or in mixtures, as the propellant. It is desirable to use it in the proportion of 1–20 wt %, i.e., such that the internal pressure becomes 2.0–6.0 kg/cm² G.

In the hair cosmetic of this invention one can also add oils and fats for cosmetic use, within limits such that the effectiveness of this invention is not harmed (0.1–10 wt %). Examples of these oils and fats are glycerides, such as castor oil, cacao oil, mink oil, avocado oil, or olive oil; waxes, such as beeswax, whale tallow, lanolin, or carnauba wax; alcohols, such as cetyl alcohol, oleyl alcohol, hexadecyl alcohol, lauryl alcohol, stearyl alcohol, isostearyl alcohol, 2-octyl dodecanol, and polypropylene glycol; esters, such as isopropyl myristate, hexyl laurate, cetyl lactate, propylene glycol monostearate, oleyl oleate, hexadecyl 2-ethylhexanoate, and octyldodecyl myristate; and silicone derivatives, such as dimethylpolysiloxane, methylphenylpolysiloxane, polyether-modified silicone oil, epoxy-modified silicone oil, amino-modified silicone oil, alkyl-modified

silicone oil, etc. Furthermore, emulsifiers can be added in order to emulsify and stabilize these fats and oils. Examples of these emulsifiers are nonionic surface active agents such as polyoxyethylene alkyl ethers. It is desirable to use ones with HLB values of 10-20 at 0.1-5 wt %, because the emulsion will be stabilized.

If desired, one can also add other suitable additives to the hair cosmetic of this invention, such as moistening agents (glycerol, propylene glycol, etc.), preservatives (parabens, etc.), thickeners (water-soluble polymers, etc.), coloring agents (dyes, pigments, etc.), and various compound fragrances, etc.

Operation and Effects of the Invention

The amphoteric polymers used in this invention have so-called "salt introduction" activity; it appears that they are dissolved in the product state, but when they are applied to the hair and then rinsed with water, the salt concentration is reduced, so that they become insoluble and adhere to the hair, showing a setting function.

The hair cosmetic of this invention has an excellent ability to form and hold hair styles, as well as an excellent effect of imparting drying ability, smoothness, softness, and anti-static properties to the hair during the drying process. It effectively protects the hair from the heat of the dryer and because it does not have an unpleasant feeling of stickiness, etc., it can be expected to have a wide range of uses for both men and women of all ages.

Working Examples

Next, this invention will be explained by giving working examples as well as examples of syntheses of amphoteric polymers.

Furthermore, the functional evaluations and measurements of the combing force and the quantity of static electricity produced in the working examples were performed by the following methods.

(1) Functional evaluations

Twenty grams of hair (15 cm long) from Japanese women who had not been given treatments such as cold perms, breeches,² etc., were bundled together and 2 g hair rinse were applied uniformly to this hair bundle. Next, the hair was rinsed out with running water for 30 seconds, after which it was dried with a towel. The softness and oily feel of the hair bundle in this damp state were evaluated functionally. The evaluations were shown by the following symbols: especially good:

²Original "buriichi" - English spelling could not be confirmed. - Translator's note

O; good O; unchanged Δ ; inferior X.

(2) Combing force

A hair bundle in the damp state, treated in the same way as in (1) above, was combed as is (containing approximately 0.7 g water per gram of hair) or after being dried for approximately 5 minutes in a dryer (containing approximately 0.1 g water per gram hair), and the load of passing the comb through the hair was measured with a strain gauge. The measurement was performed in an isothermal chamber with a temperature of 20°C and a humidity of 65%; the measurement was repeated 20 times, and the mean value (g) was used.

(3) Amount of static electricity produced

The hair bundle in the damp state mentioned above was combed 10 times in an isothermal chamber with a temperature of 20°C and a humidity of 65%, and the quantity of static electricity produced in the hair (in kV) was measured.

Synthesis Example 1

143.6 Grams water, 34.4 g dimethylaminoethyl methacrylate, and 18.9 g methacrylic acid were charged into a 300-milliliter separable flask with a stirrer, thermometer, reflux cooler, and dropping funnel attached. Then, 0.063 g ammonium persulfate and 0.026 g sodium hydrogen sulfite were added, the temperature was raised, and polymerization was performed for 5 hours at 35°C, after which curing was performed for 30 minutes at 65°C. Next, the result was introduced into a large quantity of acetone and an amphoteric polymer was obtained.

Synthesis Example 2

Four hundred sixty milliliters hexane containing 3.0 w/v % sorbitan monostearate were charged into a 1-liter separable flask with a stirrer, thermometer, reflux cooler, and dropping funnel attached, and heating and dissolution were performed at 60°C. Next, 60 g water, 50.0 g 2-acrylamide-2-methylpropanesulfonic acid, 37.9 g dimethylaminoethyl methacrylate, and 0.09 g 2,2'-azobis(2-amidinopropane) dihydrochloride were charged in. The temperature was raised to 62°C and polymerization was performed for 8 hours. After cooling was performed, the hexane was removed by decanting and the result was vacuum-dried to obtain an amphoteric polymer.

Synthesis Example 3

One hundred fifty grams water, 45.4 g methacryloxyethyltrimethyl ammonium chloride, 39.9 g sodium p-styrenesulfonate, 0.063 g ammonium persulfate, and 0.026 g sodium hydrogen sulfite were added to a 500-milliliter separable flask with a stirrer, thermometer, reflux cooler, and dropping funnel attached; the temperature was raised, and polymerization was performed for 5 hours at 35°C. After this, curing was performed for 30 minutes at 65°C. Next, the result was introduced into a large quantity of acetone and an amphoteric polymer was obtained.

Synthesis Example 4

One hundred grams water, 50.0 g methacrylamide propyl trimethyl ammonium chloride, 48.5 g potassium 3-methacrylpropanesulfonate, and 1.0 g sodium persulfate were added to a 500-milliliter separable flask with a stirrer, thermometer, reflux cooler, and dropping funnel attached. Polymerization was performed for 9.5 hours at a temperature of 65°C; after cooling, the result was introduced into a large quantity of acetone and an amphoteric polymer was obtained.

Synthesis Example 5

Five grams 3-dimethyl(methacroyloxyethyl) ammonium propane sulfonate, 20 g water, and 0.025 g potassium persulfate were charged into a 50-milliliter separable flask with a stirrer, thermometer, reflux cooler, and dropping funnel attached. Polymerization was performed for 10 hours at a temperature of 65°C. The result was introduced into methanol and an amphoteric polymer was obtained.

Synthesis Example 6

An amphoteric polymer was obtained by performing a reaction in the same manner as in Synthesis Example 5, using 5 g 3-dimethyl(methacroylamidepropyl) ammonium propane sulfonate, 20 g water, and 0.025 g potassium persulfate, and an amphoteric polymer was obtained.

In the following working examples, the performances of the hair cosmetics were evaluated by the following methods. The amphoteric polymers of Synthesis

Examples 1-6 which were used were all soluble in a 10 wt % aqueous solution of sodium chloride and insoluble in water when used at a concentration of 0.1 wt %.

Working Example 1

The compositions shown in Table 1 were prepared and their external appearances and rinsing performances were investigated.

Table 1

| Ingredient | (wt %) | |
|-----------------------------------------------------------------------|-----------------------------------|-----------------------------------|
| | Comparison Product 1 | Product of This Invention 1 |
| (1) Di-2-octyldodecyldimethyl ammonium chloride ^{*1} | - | 2.0 |
| (2) Dicotostearyldimethyl ammonium chloride | 2.0 | - |
| (3) Cetostearyltrimethyl ammonium chloride | 2.0 | 2.0 |
| (4) Steary alcohol | 2.0 | 2.0 |
| (5) Ampholytic polymer (polymer of Synthesis Example 1) ^{*2} | 2.0 | 2.0 |
| (6) Sodium chloride | 3.0 | 3.0 |
| (7) Polyoxyethylene lauryl ether (20 E.O.) | 10.0 | 10.0 |
| (8) Propylene glycol | 1.0 | 1.0 |
| (9) Purified water | 5.0 | 5.0 |
| | 75.0 | 75.0 |
| External appearance | White emulsion (dispersed system) | White emulsion (dispersed system) |

^{*1}: Synthesized from Guerbet alcohol (Enjiekoru 200A, Shin Nippon Rikasei Co.)

^{*2}: Almost the same evaluations were obtained when the polymers of Synthesis Examples 2-6 were used.

Method of preparation of compositions:

Ingredients 1, 3, 4, 7, and 8, or ingredients 2, 3, 4, 7, and 8, were heated to 70°C and dissolved, after which they were added to a mixed aqueous solution of ingredients 5, 6, and 9, heated to the same temperature; stirring and emulsification were performed, after which the result was cooled to room temperature while stirring and hair rinse compositions were obtained.

These compositions were used by 20 members of a panel of experts, and their rinsing performances were compared. The results are shown in Table 2.

Table 2

| | Comparison Product 1 better | Both products the same | Product of This Invention 1 better |
|-------------------------------------|-----------------------------------|---------------------------|------------------------------------------|
| Little oily feel | 1 | 2 | 17 |
| Softness of hair | 2 | 5 | 13 |
| Good combing ability (when damp) | 0 | 2 | 18 |
| Good combing ability (when dry) | 2 | 4 | 14 |
| Little hair fly | 4 | 5 | 11 |

Working Example 2

The compositions shown in Table 3 were prepared and their external appearances and rinsing performances were investigated.

Method of preparation of compositions:

Ingredients 2, 3, 4, 7, and 8, or ingredients 1, 2, 3, 4, 7, and 8, were heated to 70°C and dissolved, after which they were added to a mixed aqueous solution of ingredients 5, 6, and 9, heated to the same temperature; stirring and emulsification were performed, after which the result was cooled to room temperature while stirring and hair rinse compositions were obtained.

Results:

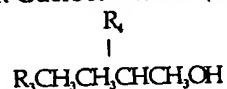
Shown in Table 3.

Table 3

| Ingredient | | Product of This Invention 2 | Product of This Invention 3 | Product of This Invention 4 |
|----------------------------------------------------------------------|-------------------------------------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| (1) Di-2-octyldodecyldimethyl ammonium chloride ¹ | | — | — | 0.1 |
| (2) 2-Dodecylhexadecyltrimethyl ammonium chloride ² | | 0.05 | 1.0 | 1.0 |
| (3) Monostearyltrimethyl ammonium chloride | | 0.2 | 0.2 | 0.2 |
| (4) Cetostearyl alcohol | | 2.0 | 2.0 | 2.0 |
| (5) Ampholytic polymer (polymer of Synthesis Example 1) ³ | | 2.0 | 2.0 | 2.0 |
| (6) Sodium chloride | | 8.0 | 8.0 | 8.0 |
| (7) Polyoxyethylene stearyl ether (20 E.O.) | | 1.5 | 1.5 | 1.5 |
| (8) Propylene glycol | | 5.0 | 5.0 | 5.0 |
| (9) Purified water | | 81.25 | 80.3 | 80.2 |
| External appearance | | White emulsion (dispersed system) | White emulsion (dispersed system) | White emulsion (dispersed system) |
| Rinse performance | (1) Functional evaluation Little oily feel Smoothness | O O | O O | O O |
| | (2) Combing force (g) Damp hair Dry hair | 170 105 | 155 98 | 148 95 |
| | Quantity of static electricity produced (kV) | 1.5 | 1.4 | 1.3 |

*1: Synthesized from Guerbet alcohol (Enjiekoru 200A, Shin Nippon Rikasei Co.)

*2: Synthesized from Guerbet alcohol (branched chain alcohol shown by



(total carbon number 28)

*3: Almost the same evaluations were obtained when the polymers of Synthesis Examples 2-6 were used.

Working Example 3

A hair rinse with the composition shown below was prepared and a comparative functional evaluation with product 3 of this invention, shown in Table 3, was performed by an expert panel of 20 members. The results are shown in Table 4.

Comparison Product 2

| | |
|-----------------------------------------------------|------------|
| Monostearyltrimethyl ammonium chloride | 1.5 (wt %) |
| Cetostearyl alcohol | 2.0 |
| Purified water | 80.0 |
| Amphoteric polymer (polymer of Synthesis Example 1) | 2.0 |
| Sodium chloride | 8.0 |
| Polyoxyethylenestearyl ether (20 E.O.) | 1.5 |
| Propylene glycol | 5.0 |

Table 4

| | Comparison Product 2 better | Both products the same | Product of This Invention 3 better |
|-------------------------------------|-----------------------------------|---------------------------|------------------------------------------|
| Little oily feel | 3 | 3 | 14 |
| Softness of hair | 2 | 4 | 14 |
| Good combing ability (when damp) | 0 | 3 | 17 |
| Good combing ability (when dry) | 4 | 5 | 11 |
| Little hair fly | 2 | 6 | 12 |

Working Example 4

Hair rinse composition:

| | |
|------------------------------------------------------------|----------------|
| (1) Di-2-hexyldecyldimethyl ammonium chloride ³ | 1.0 |
| (2) Monostearyltrimethyl ammonium chloride | 1.0 |
| (3) Cetostearyl alcohol | 2.0 |
| (4) Paraffin | 2.0 |
| (5) Amphoteric polymer (polymer of Synthesis Example 2) | 2.0 |
| (6) Polyoxyethylenestearyl ether (20 E.O.) | 1.0 |
| (7) Methyl paraben | 0.2 |
| (8) Sodium chloride | 10.0 |
| (9) Fragrance | 0.3 |
| (10) Propylene glycol | 7.0 |
| (11) Water | <u>Balance</u> |
| | 100.0 wt % |

Method of preparation:

Ingredients 5, 7, and 8 were homogeneously dispersed in 11 and the result was heated. A homogeneous, heated solution of 1, 2, 3, 4, and 10 was added, while stirring was performed. After the result was cooled, 9 was added, and a hair rinse composition which imparted a good feel and styling ability to hair was obtained.

Working Example 5

Hair rinse composition:

| | |
|---------------------------------------------------------------|-----|
| (1) Dicotostearyldimethyl ammonium chloride | 1.0 |
| (2) 2-Decyltetradecyltrimethyl ammonium chloride ⁴ | 1.0 |

³Synthesized from Guerbet alcohol (Enjiekoru 160B, Shin Nippon Rikasei Co.)

⁴Synthesized from Guerbet alcohol (Enjiekoru 240A, Shin Nippon Rikasei Co.)

| | |
|---------------------------------------------------------|----------------|
| (3) Cetyltrimethyl ammonium chloride | 0.5 |
| (4) Behenyl alcohol | 2.0 |
| (5) Dimethylpolysiloxane | 1.0 |
| (6) Amphoteric polymer (polymer of Synthesis Example 1) | 3.0 |
| (7) Polyoxyethylenebehenyl ether (10 E.O.) | 1.5 |
| (8) Methyl paraben | 0.2 |
| (9) Xanthan gum | 0.5 |
| (10) Sodium chloride | 8.0 |
| (11) Fragrance | 0.5 |
| (12) Propylene glycol | 10.0 |
| (13) Water | <u>Balance</u> |
| | 100.0 wt % |

Method of preparation:

Ingredients 6, 8, 9, and 10 were homogeneously dispersed in 13 and the result was heated. A homogeneous, heated solution of 1, 2, 3, 4, 5, and 7 was added, while stirring was performed. After the result was cooled, 1 was added, and a hair rinse composition which imparted a good feel and styling ability to hair was obtained.

Working Example 6

Hair conditioning mousse composition:

| | |
|------------------------------------------------------------------------------------------------------|-----|
| (1) Di-2-hexyldecyldimethyl ammonium chloride ⁵ | 0.5 |
| (2) 2-Dodecylhexadecyltrimethyl ammonium chloride (the same as ingredient 2 of Working Example 2) | 0.3 |
| (3) Cetyltrimethyl ammonium chloride | 1.0 |
| (4) Poxoxyethylenehexadecyl ether (20 E.O.) | 0.5 |

⁵Synthesized from Guerbet alcohol (Enjiekoru 160B, Shin Nippon Rikasei Co.)

| | |
|---------------------------------------------------------|----------------|
| (5) Lecithin | 0.2 |
| (6) Amphoteric polymer (polymer of Synthesis Example 3) | 1.5 |
| (7) Sodium chloride | 5.0 |
| (8) Propylene glycol | 1.0 |
| (9) Ethanol | 5.0 |
| (10) Fragrance | 0.3 |
| (11) Propellant (LPG) | 7.0 |
| (13) Water | <u>Balance</u> |
| | 100.0 wt % |

Method of preparation:

Ingredients 6 and 7 were homogeneously dispersed in 12 and the result was heated. A homogeneous, heated solution of 1, 2, 3, 4, 5, 8, 9, and 10 was added, while stirring was performed. The result was loaded into a pressure-resistant aerosol container and it was filled with the propellant to obtain a hair conditioning mousse composition.

Working Example 7

Hair conditioning mousse composition:

| | |
|------------------------------------------------------------------------------------------------------|-----|
| (1) Dialkyldimethyl ammonium chloride ⁶ | 0.5 |
| (2) 2-Dodecylhexadecyltrimethyl ammonium chloride (the same as ingredient 2 of Working Example 2) | 1.0 |
| (3) Polyoxyethyleneoctyldodecyl ether (20 E.O.) | 1.0 |
| (4) Siracanba [Betula platyphylla Sukatcev var] oil extract | 0.5 |
| (5) Amphoteric polymer (polymer of Synthesis Example 3) | 1.0 |
| (6) Sodium chloride | 8.0 |

⁶Branched quaternary ammonium salt derived from commercial alcohols with carbon numbers of 12-15 synthesized by the oxo method (mixture of equal quantities of Dobanoru 23 and Dobanoru 45, Mitsubishi Petrochemical Co.); its branching rate was 20 wt %.